UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

5/27/88

MEMORANDUM

FROM: Connie B. Welch, Chemist
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Registration Division (TS-767C)

TO: Richard F. Mountfort, PM 23
Fungicide-Herbicide Branch
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THRU: Lynn M. Bradley, Head
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Registration Support and
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THRU: Ferial S. Bishop, Chief
Registration Support and
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Registration Division (TS-767C)

REQUESTOR: Hoechst Celanese

REGISTRATION NOS.: 8340-EO Ignite, Technical — TGAI
8340-EI Ignite, Herbicide — EP
8340-ET Ignite, Manufacturing-Use Product — MP

MRID No.: 40345501-03; Accession No.: 263025
Pesticide Chemical Code: 128850-5
Company Code: HOE-039866
CAS Registry No.: 77182-82-2
Chemical Name: Monoammonium 2-amino-4-(hydroxymethylphosphinyl) butanoate
Common/Trade Name: Glufosinate-ammonium, Basta, Ignite
Use: Herbicide

Introduction

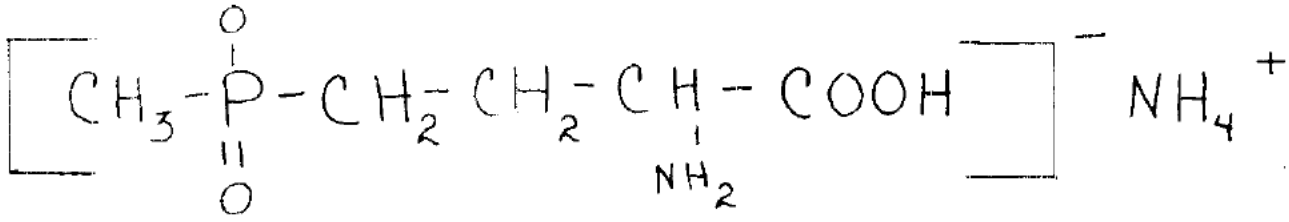
This submission is for the manufacturing-use product (MP), end-use product (EP), and technical containing the active ingredient (AI) monoammonium 2-amino-4-(hydroxymethylphosphinyl) butanoate. All product chemistry requirements pertaining to manufacturing use products and end use products as described in the Code of Federal Regulations, Volume 40, Section 158.120 must be met to achieve full registration.

Product chemistry for Ignite, Technical was reviewed by John M. Worthington of the Residue Chemistry Branch, Hazard Evaluation Division, in conjunction with a temporary tolerance for Ignite on January 2, 1985.

Product Identity and Composition

61-1 - Product Identity and Disclosure of Ingredients

Monoammonium 2-amino-4-(hydroxymethylphosphinyl)butanoate is the name of the active ingredient found in the manufacturing use product, end use product, and technical produced by the Hoechst Celanese Corporation.



Other identifying names include: Butanoic acid, 2-amino-4-(hydroxymethylphosphinyl)-, monoammonium salt; ammonium-dl-homoalanin-4-yl(methyl)phosphinate.

Empirical Formula: $C_5H_{15}N_2O_4P$
Molecular Weight: 198.1
CAS Registry No.: 77182-82-2
Pesticide Chemical Code: 128850-5

Information regarding the identity of the impurities for Ignite, Technical are given in the Confidential Statement of Formula (CSF). Information regarding the intentionally added inert ingredients, which are cleared for food use, for the end use product Ignite, Herbicide are also given. Refer to Confidential Appendix B for the disclosure of these ingredients.

No CSF was submitted for the manufacturing use product, only a list of the impurities was given. Refer to Confidential Appendix A for the disclosure of these impurities.

61-2 - Description of Beginning Materials and Manufacturing Process

Refer to Confidential Appendix A for a description of the beginning materials and manufacturing process for Ignite, Technical and Manufacturing Use Product. The following information was not submitted:

A. Beginning Materials

1. Copies of all available technical specifications, data sheets, and other documents by which the composition, properties, or toxicity are described;
2. All other information concerning qualitative and quantitative composition of the beginning materials.

B. Manufacturing Process

1. Statement of whether the process is a batch or continuous process;

2. Information on the relative amounts and the order in which they were added;

3. Description of equipment used to produce the product;

4. The duration of each process;

5. A description of any physical conditions which are controlled during each step; and

6. Description of the purification procedures including procedures to recover or recycle starting materials, intermediates, or final product.

No manufacturing or formulation process is given for the end use product. Information should be submitted stating whether the process involves any chemical reactions or just a mixture of the technical product and inert ingredients. If no reactions occur, the formulating procedure should be described.

61-3 - Discussion of the Formation of Impurities

Refer to Confidential Appendix A for a discussion of the formation of impurities for the technical and manufacturing use product. No discussion is given of any impurities formed in the end use product.

Analysis and Certification of Product Ingredients

62-1 - Preliminary Analysis

Ignite, Technical

The applicant has submitted the results of five representative samples of Ignite, Technical, which were analyzed for the active ingredient and each impurity. Refer to Confidential Appendix B for the results of the analyses. The following additional information is required:

1. A complete detailed description of each step in the analytical method, AL 42/32;

2. The mean and standard deviation of the reported results; and

3. A statement of the precision and accuracy of the analytical method.

Manufacturing Use Product

The technical grade active ingredient (TGAI) is separated from impurities and other ingredients by high performance liquid chromatography (HPLC) on a strongly basic anion exchange column, 125 x 4.6 mm. The mobile phase consisted of a potassium dihydrogenphosphate solution C (KH_2PO_4) with a molar concentration of 0.1 mol/L in water. It is detected spectrophotometrically by UV absorption at a wavelength of 195 nm.

Sample and standard solutions are prepared as aqueous solutions of the precise amounts of the sample. The amount is determined with a precision of at least 0.1 mg. The evaluation is done by the external standard method; therefore, sample injection is carried out by means of a sample loop injector using the "completely filled loop" mode. A second standard solution with a concentration at least 10 percent different from the first is injected as a means of checking the correct weighout of the initial standard solution and the linear range of the instrument. The constant response factor is also checked.

To determine the precision of the analytical method, a typical sample of the TGAI was analyzed six times. The mean

value was 19.85 percent with a standard deviation of 0.71 percent. The accuracy was determined by analyzing a total of five samples consisting of a small amount of a blank formulation of typical composition and a precise amount of pure sample. The mean recovery of the spiked blank formulation was 99.95 percent with a standard deviation of 0.82 percent. The linear range for the analytical method was 66 to 330 mg/100 mL with less than 1.5 percent deviation.

62-2 - Certification of Limits

The applicant has submitted a CSF (EPA Form 8570-4), which includes the certified limits for Ignite, Technical and the end use product Ignite, Herbicide. No limits were submitted for the manufacturing use product. Refer to Confidential Appendix B for the disclosure of these limits.

62-3 - Analytical Methods to Verify Certified Limits

Analytical methods and test results were submitted for five representative samples of Ignite, Technical, which were analyzed for the active ingredient and each impurity. The methods used for the analysis are given below.

Substance	Method of Detection
Active Ingredient	AL 42/32
Methanol	GC
1-Butanol	GC
Water	KF
NH_4^+ (Surplus)	Kjeldahl
Chloride, Cl^-	Titration
Acetate	Ion Chromatography

A description of the analytical methods for the determination of ammonium and chloride in Ignite, Technical was given. Both are determined according to the AOAC Official Methods of Analysis. Ammonium is determined using method 2.065 and chloride is determined using method 34.128(a). However, no description was given of the method AL 42/32 used to detect the active ingredient. Therefore, the data submitted cannot be acceptable until a complete description of the method is provided, as requested under 62-1.

The method submitted to verify the certified limits for the end use product is HPLC. Ignite is separated from its impurities using a strongly basic anion exchange column and is detected spectrophotometrically by measurement of UV absorption at a wavelength of 200 nm. The quantitative comparison is carried out by comparison with an external standard.

A sevenfold determination of the technical grade active ingredient yielded a mean value and standard deviation of 94.5 ± 0.4 percent absolute (0.4% relative).

Physical and Chemical Characteristics

Summarized below are several physicochemical properties of the manufacturing use product, end use product, and active ingredient as furnished by the applicant.

Guidelines Reference No. (40 CFR 158.120) and Name of Property	Description
63-2 - Color	PAI - White TGAI - White to light brown MP - Colorless to light brown EP - Blue to bluish green
63-3 - Physical State	PAI - Crystalline powder TGAI - Crystalline powder MP - Liquid at normal temperatures EP - Liquid at normal temperatures
63-4 - Odor	PAI - Weakly pungent TGAI - Odorless to weakly pungent MP - Slightly pungent EP - Weakly pungent
63-5 - Melting Point	PAI - 488° K (215° C) under decomposition MP - NR EP - NR
63-6 - Boiling Point	TGAI & Pure Sub - Not determinable because of decomposition of the AI at its melting point. MP - NR EP - NR
63-7 - Density, Bulk Density, or Specific Gravity	PAI & TGAI - 1.4 g/cm^3 (20° C) MP - 1.20 ± 0.005 g/cm^3 (20° C) EP - 1.11 ± 0.05 g/cm^3 (20° C)
63-8 - Solubility	TGAI - 1370 g ± 11/1000 mL water (22° C) PAI - 65 mg/100 mL Ethanol 16 mg/100 mL Acetone 14 mg/100 mL Toluene 20 mg/100 mL N-Hexane

	14 mg/100 mL Ethyl acetate MP - NR EP - NR
Guidelines Reference No. (40 CFR 158.120) and Name of Property	Description
63-9 - Vapor Pressure	TGAI - Not determinable because of decomposition of AI by heating. MP - NR EP - NR
63-10 - Dissociation Constant	TGAI - $pka_1 < 2$; $pka_2 = 2.9$; $pka_3 = 9.8$ MP - NR EP - NR
63-11 - Octanol/Water Partition Coefficient	AI - $p < 0.1$ MP - NR EP - NR
63-12 - pH	PAI - 4.7 ± 1 MP - 5.5 - 6.5 EP - 5.6 ± 1 (1% solution in distilled water) 5.7 ± 1 (10% solution in distilled water)
63-13 - Stability	AI - Stable for at least 8 days at room temperature. EP - Heat stability - If stored in a sealed glass bottle at 50° C for 3 months, the product remains chemically and physically stable. EP - Cold Stability - Formulation does not separate when maintained at 0° C for 4 weeks.
63-14 - Oxidizing or Reducing Action	MP - NR EP - NR
63-15 - Flammability	MP - N/A (50% water) EP - 65 ± 2° C (open) > 100° C (closed) TGAI - Not determinable because of an exothermic decomposition of the AI at the melting range.

Guidelines Reference No. (40 CFR 158.120) and Name of Property	Description
63-16 - Explodability	MP - N/A (50% water) EP - Not explosive material TGAI - Not capable of dust explosion
63-17 - Storage Stability	MP - Stable when stored in glass bottle at temperatures of 50° C for 3 months. If stored in unopened original containers at temperatures of 25° ± 5° C, remains physically and chemically stable for at least 2 years. TGAI - Remains physically and chemically stable for 2 years if stored in unopened original containers at 25° ± 5° C; or for 3 months at 50° ± 1° C. EP - Remains chemically and physically stable for 2 years if stored in original containers at 25° ± 5° C. MP - 44 ± 4 mPa.s (cp) 20° C EP - 68 ± 5 mPa.s (cp) 20° C
63-19 - Miscibility	MP - NR EP - Miscible w/water, ketones, and alcohols.
63-20 - Corrosive Characteristics	TGAI - Not corrosive to polyethylene bags used as packing material. EP - Not corrosive to glass bottles or high-density polyethylene containers used as packing material.
63-21 - Dielectric Breakdown	EP - Not for use around electrical equipment.

Recommendation

The product chemistry data requirements for the manufacturing use product, end use product, and technical have not been completely satisfied. The following additional information is required before registration can be issued:

1. Series 61-2 - Description of Beginning Materials and Manufacturing Process (Ignite, Technical and MP)

   Copies of all available technical specifications, data sheets, and all other information concerning qualitative and quantitative composition of the beginning materials; statement of whether process is a batch or continuous process; information on relative amounts and the order in which they were added; a description of the equipment used; the duration of each process and a description of any physical conditions which are controlled; and a description of any purification procedures;

2. Series 61-2 - Description of Beginning Materials and Manufacturing Process (Ignite, Herbicide)

   A detailed description of the manufacturing process;

3. Series 61-3 - Discussion of the Formation of Impurities

   A complete description of any impurities formed during the manufacturing process;

4. Series 62-1 - Preliminary Analysis - MP and TGAI

   A complete detailed description of the analytical methods used to detect the active ingredient (AL 32/42) and the impurity methanol; the mean and standard deviation of the reported results;a statement of the precision and accuracy of the methods;

5. Series 62-2 - Certification of Limits

   A CSF for the manufacturing use product; and

6. Series 62-3 - Analytical Methods to Verify Certified Limits

   A description of the analytical method used to obtain certified limits for the manufacturing use product.

As required for a pesticide registration, samples of the technical grade active ingredient (200 g) and pure active ingredient (5 g) should be submitted along with the analytical method for the active ingredient to the following address:

Active Ingredients Program
Attn: Head, Analytical Chemistry Section
Chemical Operations Branch
Benefits and Use Division
Office of Pesticide Programs
Environmental Protection Agency
Building 306, BARC East
Beltsville, MD 20705

Attachments

Confidential Appendix A
Confidential Appendix B
Product Chemistry Information Form A
Product Chemistry Information Form B

Attachment I

PRODUCT CHEMISTRY INFORMATION FORM A

1. Product Name: Ignite
2. EPA Reg. No. (if known): 8340-EO, EI, ET
3. Registrant Name: Hoechst Celanese
4. Active Ingredient(s): monoammonium 2-amino-4-hydroxy-methylphosphinyl)butanoate

INFORMATION REQUESTED OF REGISTRANT	DATE Req.	DATE Rec.
1. Safety, Handling, & Storage Data		
2. Analytical Method for Active Ingredient (ai)	X	
3. Analytical Method for Specific Impurity	X	
4. Analytical Grade Standard of ai	X	
5. Analytical Standard of Specific Impurity*		
6. Technical Grade of ai*	X	
7. MUPs Grade Material with ai*		
8. EUPs Grade Material with ai*		
9. Melting Point		
10. Boiling Point		
11. Solubility of ai		
12. Spectral Data, IR		
MS		
NMR		
13. Chromatographic Data, GLC		
14. Other		

*With Purity Statement

Attachment II

PRODUCT CHEMISTRY INFORMATION FORM B

Request for Laboratory Evaluation

1. EPA Reg. No. 8340-EO, EI, ET
2. RD File No. _____
3. ACS Lab No. _____
4. Date Request Sent to ACS _____ _____
5. Date Received by ACS _____ _____
6. Date Assigned _____ _____
7. Date of Followup _____ _____
8. Date Reported to RD _____ _____

To be sent when method received

INFORMATION REQUESTED OF ACS	DATE Req.	DATE Rec.
1. Validation of Active Ingredient Method	X	
2. Validation of Method for Specific Impurity		
3. Validation of Melting Point		
4. Validation of Boiling Point		
5. Validation of Spectral Data, IR		
MS		
NMR		
6. Validation of Chromatographic Data, GLC		
HPLC		
7. Other		

COMMUNICATION LOG ON REVERSE

Pages 13-18 Manufacturing process information may be entitled to confidential treatment